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(54) SPECTROPHOTOMETER

(57) Abstract:

PURPOSE: To measure an attenuated total reflection spectrum without transmitting a sample through a prism by forming a polymer film which holds the sample on the surface of the prism and does not allow the sample to be transmitted through the prism.

CONSTITUTION: After a polymer film 22 is closely stuck to the surface of an attenuated total reflection prism 21, a sample 23 is introduced onto the film 22. The sample 23 is prepared by dissolving, for example, a high molecular component 24 such as a protein, etc., like the blood and low molecular component 25, such as urea, etc., in water. Since the polymer film 22 is constituted of a water-soluble polymer material having a large number of hydrophilic groups, such as crosslinked polyacrylic soda, the water contained in the sample and components 24 and 25 dissolved in the water can be sucked in the film 22 by the affinity between polymer ions and water. Moreover, the light 26 made incident to the end face of the prism 21 is propagated through the prism 21 by total reflection and forms a spectrum, because a specific wavelength of the light is absorbed when the light is totally reflected by the interface of the prism 21.

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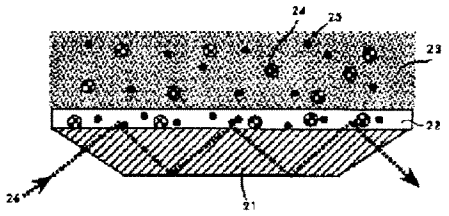

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application]The spectrophotometer of this invention is related with the device which analyzes the organic substance in solution by infrared spectroscopy and attenuated total reflection spectroscopy.
[0002]
[Description of the Prior Art]About analysis of the liquid component by the combination of the conventional attenuated total reflection spectroscopy and infrared spectroscopy, Analytical In the 2023rd page (Analitical Chemstry, 61, and (1989) pp.2016-2023), it has discussed in the chemistry 61 and (1989) from the 2016th page. Here, the biochemistry ingredient of whole blood and plasma is performed using the optical cell and Fourier transform infrared spectrophotometer which used the attenuated-total-reflection prism of rod-like zinc selenide. In a thing related as this kind of a device, it is applied. In the 1646th page (Applied Spectroscopy, 44 (1990), pp.1641-1646), it has discussed in spectroscopy 44 (1990 years) from the 1641st page. Here, the influence of measurement on the water which analyzes the organic matter in solution using the attenuated-total-reflection prism which carried out the coat of the polymer, and is contained in a sample as a result is removed, and it is being argued that the detection limit improved. JP,55-500589,A discusses the inner total-reflection-spectrum method which uses a transparent high refractive index substance as an exchangeable interlayer between reflection elements and a sample, in order to reproduce the beautiful surface optically [reflection elements].
[0003]
[Problem(s) to be Solved by the Invention]The adsorption of the polymeric component which consideration is not carried out about adsorption of the sample component to the attenuated-total-reflection prism surface, and cannot remove the above-mentioned conventional technology easily especially due to washing caused contamination of a sample, and an error of measurement of the spectrum. This invention reduces polymers adsorption on the attenuated-total-reflection prism surface, and an object of this invention is

to aim at improvement in analysis accuracy.

[0004]Consideration was not carried out about exchange of the film which intervenes between attenuated-total-reflection prism and a sample, an interlayer, and a sample, but the above-mentioned conventional technology had low pressure of business nature. It aims at measuring an attenuated-total-reflection spectrum, without enabling interlayer exchange of one-touch and making a sample penetrate on prism in this invention.

[0005]

[Means for Solving the Problem]To achieve the above objects, a poly membrane which is not made to penetrate on attenuated-total-reflection prism, holding a sample on the attenuated-total-reflection prism surface is formed.

[0006]

[Function]The poly membrane which consists of water soluble polymers sticks and exists on attenuated-total-reflection prism. The sample which used water as the solvent is dropped on this poly membrane. A poly membrane is what constructed the bridge and formed the water soluble polymer in the network structure, and absorbs and gels moisture from a sample. Under the present circumstances, an objective component is diffused in gel together, when moisture permeates hydro-gel out of a sample. Since such hydro-gel does not emit water outside to the external pressure, it can measure a spectrum, without polluting the surface of attenuated-total-reflection prism by a sample.

[0007]The poly membrane which carried out water beforehand can form hydro-gel, and can control surface adsorption of the polymeric component contained in a sample.

[0008]

[Example]This invention is explained in detail based on an example below. Drawing 1 is a lineblock diagram of the Fourier transform infrared spectrophotometer (Fourier Transform Infrared Spectroscopy, FT-IR) which is the first example of this invention. The light emitted from the light source 1 receives abnormal conditions with Michelson interferometer 2, and is emitted as an interferogram. the interferometer 2 to emitted light -- ATR prism 4 for reference of the sample chamber 3 -- or it enters into one of ATR prisms 5 for samples. The light which penetrated prism is changed into an electrical signal by the detector 6, and after being amplified, it is downloaded to the computer department 8 through A/D converter 7. If only a predetermined number finishes sampling one interferogram of the object for reference, or the ATR prism for samples, the optical path of a sample chamber will be changed and measurement of the remaining prism will be performed similarly. Drawing 2 is a strabism sectional view of the optical cell circumference of this example. On ATR prism 4 for reference, and ATR prism 5 for samples, adhesion immobilization of the poly membrane 9 for its ****** samples and the poly membrane 10 for samples is carried out by the frame 11. The sample for reference and a test portion are dropped on the poly membrane 9 and 10, respectively, and an interferogram is measured. Drawing 3 is a flow chart of the measurement procedure of this example. Each interferogram of a reference sample and a test portion is measured, and it changes into (12) and a spectrum (13). The common

characteristic absorption band of a reference spectrum and a sample spectrum is searched, it integrates with the characteristic absorption band of (14) and each spectrum, and (15) and a difference coefficient are computed (16). What multiplied the reference spectrum by the difference coefficient is deducted from a sample spectrum, and a differential spectrum is formed (17). If it integrates with the differential spectrum of the same wave number field as the above-mentioned characteristic absorption band and (18) and an integral value are 0 (19), (20 which display a differential spectrum as it is). If an integral value is not 0, the operations from 14 to 18 will be similarly performed by using the differential spectrum as a sample spectrum.

[0009]Drawing 4 is a sectional view of the fundamental composition of the optical cell portion of this invention. ATR prism 21 and the poly membrane 22 are stuck, and the sample 23 is introduced on the poly membrane 22. As for the sample 23, the polymeric components 24, such as protein, and the low molecule ingredients 25, such as urea, have melted into water, for example like blood. The poly membrane 22 comprises a water soluble polymer which has many hydrophilic groups in intramolecular, for example like bridge construction sodium polyacrylate. If bridge construction sodium polyacrylate contacts water, it will dissociate to a polymer ion with negative electrification ($-COO^-$), and a low-molecular counter ion with positive electrification (-Na+). If counter ion concentration is made higher than the concentration in the surrounding blood, it will have osmotic pressure to the exterior, the counter ion will separate from the polymer ion, and a polymer ion will become difficult to be influenced by a counter ion. As a result, the chain of a polymer ion is extended by the electric repulsive force of the anions in a polymer ion, and the moisture in a sample and the ingredients 24 and 25 which are dissolving in it can be absorbed with the affinity of a polymer ion and water in a poly membrane. Since it is included in the meshes of a net of a polymers chain, neither water nor the ingredients 24 and 25 are emitted even if it applies the external pressure to the poly membrane 22. The light 26 which entered into the ATR prism 21 end face on the other hand spreads the inside of prism by total internal reflection. In the case of the total internal reflection in this prism interface, light has the specific wavelength which has sunk in into the sample holding layer about several micrometers absorbed, and forms a spectrum.

[0010]Drawing 5 is a sectional view of the optical cell part of the 2nd example of this invention. The bipolar membrane 28 which made the porous layer 27 surface carry out the surface polymerization of the water soluble polymer, and formed the poly membrane 22 in it is stuck on the ATR prism 21 surface. The porous layer 27 is formed from a polyethylene porosity film, a cellulose type filtration membrane, a polycarbonate porosity film, a polyvinyl chloride system filtration membrane, a polysulfone system filtration membrane, etc. with the surface smoothness which can make the poly membrane 22 penetrate a sample, and does not absorb a sample. The Honda porous layer 27 plays the role which reinforces the poly membrane 22 which comprises hydro-gel with a weak mechanical strength. The sample dropped at the porosity side side of the bipolar membrane 28 penetrates the porous layer

27, and is absorbed by the poly membrane 22. The light 26 which entered into the ATR prism 21 end face on the other hand spreads the inside of prism by total internal reflection. In the case of the total internal reflection in this prism interface, light has the specific wavelength which has sunk in into the sample holding layer about several micrometers absorbed, and forms a spectrum.

[0011]Drawing 6 is a sectional view of the 3rd example optical cell part of this invention. The poly membrane 22 is directly formed in the ATR prism 21 surface. An amino group, a carboxyl group, a hydroxyl group, etc. are introduced into the ATR prism 21 surface by the low-temperature plasma treatment in oxygen, nitrogen, hydrogen, and ammonia gas, With reaction reagents, such as an isocyanate, cyanuric chloride, carbodiimide, and glutaraldehyde, hydro-gel is fixed by a coupling reaction and the poly membrane 22 is formed. Or the active species in which a polymerization start is possible is introduced into the ATR prism 21 surface, the graft polymerization of the vinyl monomer is carried out to it by a gamma ray, glow discharge, ultraviolet radiation, etc., and the 1 micrometer or less-thick poly membrane 22 is formed in it. Water is beforehand carried out to the poly membrane 22, and the hydro-gel of high water content is formed. The sample 23 is introduced on it. Since there are few differences of surface energy with the sample 23 which uses the surface and water of the poly membrane 22 as the main ingredients, adsorption of a up to [the poly membrane 22 of the polymeric components 24, such as protein contained in a sample,] is controlled notably. Since the poly membrane 22 is thinner enough than the depth of the light which oozes from an ATR prism, the light 26 can sink in into the sample on a sample holding layer, and it can measure the spectrum of a sample.

[0012]Drawing 7 is a sectional view of the ATR prism polymers membrane interface of the optical cell of the 4th example of this invention. By making crosslinking density of the poly membrane 22 high, the reticulation of the meshes of a net of a polymers chain is carried out. It is contained in the sample 23 by the molecular sieve effect, and the polymeric components 24, such as protein, cannot go into the meshes of a net in the poly membrane 22, but can carry out fractionation of the polymeric component and low molecule ingredient in a sample. Measurement of low molecule ingredients, such as glucose which the polymeric component had blocked conventionally, is high-sensitivity-ized by this.

[0013]Drawing 8 is the example general drawing of this invention 1st. The main part serves as a disk top type of integral construction with which the spectroscope part 27 and the computer department 28 were put together.

The sample attaching part 9 of a cassette type is inserted in on ATR prism 21, and it is crowded, and measures by introducing a sample.

The keyboard 29 performs analytical practice and the spectrum and analysis result which were measured are outputted with the display 30 or the printer 31.

[0014]Drawing 9 is a spectrum showing the effect of the first example of this invention. The spectrum 32 is a spectrum of the ATR prism after measuring protein solution and

discarding and washing a sample by ATR/FT-IR which does not use the conventional poly membrane. The arrow 33 shows the peak of the amide which is a characteristic absorption band which protein has.
The protein in a sample sticks to the prism surface, and washing shows not falling.
On the other hand, the spectrum 34 is a spectrum of the ATR prism after measuring protein solution by FT-IR which is the first example of this invention. It turns out that a peak is not seen but there is no adsorption of a sample component in the prism surface.
[0015]Drawing 10 is a spectrum showing the effect of the 4th example of this invention. The mixed water solution of molecular weight about 130 creatinine, and the albumin of the molecular weight 10,000, The result in which the spectrum measured by ATR/FT-IR which does not use the conventional polymers measured the same sample by the spectrum 35 and ATR/FT-IR using the 4th example of this invention on the other hand is the spectrum 36. Although the influence of the characteristic absorption band of protein is looked at by the arrows 37 and 38 by the spectrum 35, by the spectrum 36, those absorption bands were not seen, but fractionation of the protein ingredient was carried out by the poly membrane, and only the creatinine which is a low molecule ingredient can be measured.
[0016]
[Effect of the Invention]According to this invention, without polluting the ATR prism surface with a sample, it can measure and there is no necessity for washing on the surface of prism. By forming hydro-gel on an ATR prism directly, the polymers adsorption in the sample on the surface of prism can be controlled, and reinforcement of prism and improvement in analysis accuracy are attained.

[Translation done.]

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CLAIMS

[Claim(s)]
[Claim 1]A spectrophotometer having the attenuated-total-reflection prism which provided a poly membrane with water absorption capacity in the surface in a device which analyzes a fluid through water by attenuated total reflection spectroscopy in an optical cell.
[Claim 2]A spectrophotometer having the attenuated-total-reflection prism which provided in the surface a poly membrane which carried out partial bridge construction and insolubilized a water soluble polymer which holds many hydrophilic radicals in intramolecular in a device which analyzes a fluid through water by attenuated total reflection spectroscopy in an optical cell.
[Claim 3]A spectrophotometer having the attenuated-total-reflection prism which provided in the surface bipolar membrane which compounded the poly membrane according to claim 1 with a porous base material in an optical cell.
[Claim 4]A spectrophotometer having the optical cell which provided a frame for fixing and sticking this poly membrane on the attenuated-total-reflection prism surface in the circumference of the poly membrane according to claim 1.
[Claim 5]A spectrophotometer with an optical cell which restricts a molecular weight of an ingredient contained in this poly membrane by changing crosslinking density of a water soluble polymer which constitutes the poly membrane according to claim 1.
[Claim 6]A spectrophotometer, wherein the poly membrane according to claim 1 is 1/3 or less thickness of wavelength of light which enters into attenuated-total-reflection prism.
[Claim 7]A coating method sticking this poly membrane to the attenuated-total-reflection prism surface established in the surface, and making the poly membrane according to claim 1 fix to it.
[Claim 8]or [that the poly membrane according to claim 1 is formed from an insolubilized water soluble polymer which is produced by constructing a bridge in Polly 2-hydroxyethyl methacrylate, polyacrylamide, Polly N-vinyl pyrrolidone, polyvinyl alcohol, starch, and carboxymethyl cellulose] -- or, A spectrophotometer forming from a complex produced by mixing these polymers to rubber or a plastic.

[Claim 9]A spectrophotometer, wherein the attenuated-total-reflection prism according to claim 1 comprises KRS-5, KRS-6, ZnSe, germanium, Si, As_2Se_3, AgCl, AgBr, sapphire, etc.

[Claim 10]A spectrophotometer provided with a mechanism which carries out automatic correction of the background and outputs a spectrum of only a sample in the spectrophotometer according to claim 1.

[Translation done.]